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# Effect of Al<sub>2</sub>O<sub>3</sub> concentration on the mechanical properties of $\gamma$ -TiAl-based alloys at high temperature

D.Pilone\*, A.Brotzu, G.Pulci, L.Paglia, F. Marra and F.Felli

*Dipartimento ICMA, Sapienza Università di Roma, Via Eudossiana 18, 00184 Roma, Italy*

## Abstract

$\gamma$ -TiAl-based alloys have been studied for many years because they are interesting candidates for the production of mechanical components working at high temperature. Increasing toughness and mechanical resistance of these alloys is crucial for boosting their application. In this paper, after having selected its composition, the alloy was produced by centrifugal casting and was reinforced by using Al<sub>2</sub>O<sub>3</sub> nano- and micro-dispersoids. Four-point bending tests performed at high temperatures highlighted that the addition of 2% of Al<sub>2</sub>O<sub>3</sub> is more effective than 3% of Al<sub>2</sub>O<sub>3</sub> in increasing the alloy mechanical properties over the 800–900 °C temperature range. The study revealed that this can be explained considering that a lower percentage of dispersoids allows to improve the oxide dispersion and to decrease the dispersed particle size.

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## 1. Introduction

Titanium aluminides are characterized by an attractive combination of properties such as low density and good oxidation resistance at high temperatures with unique mechanical properties. They are one of the few classes of novel materials that are potential candidates to be used in demanding high-temperature structural applications (Appel et al. (2000)). Over the last decades many studies have been carried out to optimize composition and microstructure of  $\gamma$ TiAl based alloys. In fact, despite their enormous potential for structural application they suffer from low ambient

\* Corresponding author. Tel.: +39 06 44585879.

*E-mail address:* [daniela.pilone@uniroma1.it](mailto:daniela.pilone@uniroma1.it)

temperature ductility and toughness (Bewlay et al. (2016)). Advances have been obtained by controlling the alloy microstructure through the use of selected alloying elements and by improving the manufacturing process and the possible thermal treatments (Ostrovskaya et al. (2021), Brotzu et al. (2020)).

The dispersion of refractory oxide phases with high elastic modulus can lead to an increase in the alloy mechanical properties as a function of the particle elastic modulus and of the optimization of the dispersed phase concentration. In particular, it is possible to obtain significant improvements of the alloy behaviour at high temperatures. This technique is extensively used for several materials called metal-ceramic particle composites. Few studies have been done on intermetallic matrix composites such as TiAl (Rittinghaus et al.(2019), Ai et al. (2008), Lu et al. (2020), Kenel et al.(2017)). In particular, the realization of these composites by casting implies significant problems due to the high density difference between the molten metal and the dispersoids and due to the particle agglomeration in the metallic matrix (Brotzu et al. (2020)). For these reasons the most used production technique for these materials is powder metallurgy and sintering. Other important aspects related to the production of metal matrix composites that must be considered concern interface structures, metal-ceramic chemical reactions, bonding strength, wettability, difference between particle and matrix thermal expansion coefficient, etc. This research follows other studies (Brotzu et al (2018), Pilone et al. (2020), Pilone et al. (2021), Pilone et al. (2022)) aimed at the realization of a TiAl- $\text{Al}_2\text{O}_3$  composite by using the centrifugal casting technique. In this case using casting as production process is an advantage because TiAl and  $\text{Al}_2\text{O}_3$  have almost the same density. Aim of this work is to evaluate the distribution of particles and the mechanical properties of two alloys containing 2% and 3% of  $\text{Al}_2\text{O}_3$ .

## 2. Experimental

The specimens necessary for the mechanical tests were produced by investment casting. For the casting a wax model was prepared by assembling 5 specimens having a size of 4.5 mm×4.5 mm×55 mm with the feeding system. A particular alumina based ceramic material was selected for the mould to avoid metal-mould reaction. The specimens were obtained by induction melting in vacuum from pure Ti, Al, Cr chips and Nb powder after six washing cycles with argon. For the tests 2% and 3% vol. of  $\text{Al}_2\text{O}_3$  was added to the alloy.  $\text{Al}_2\text{O}_3$  nanoparticles were added in the crucible. The molten metal was directly cast by using centrifugal casting into a rotating mould. After the metal solidification the mould was broken, and the casting was extracted. The specimens were then ground and polished to particular dimensions, 2 mm × 4 mm × 45 mm, in order to perform four-point bending tests at various temperatures according to the ASTM C1161 (room temperature) and ASTM C1211 (high temperature) standards, usually employed to test brittle materials. The samples were heated at 15 °C/min up to the test temperature, maintained at this temperature for 30 min and then subjected to bending tests. For each temperature, three samples were tested. The bending tests were performed with a Zwick-Roell Z 2.5 testing machine equipped with a Maytec furnace, a 3 point-contact extensometer and a silicon carbide fully articulated flexure device. In order to perform microstructural examination, specimens were observed by using optical and electron microscopes: the optical microscope was Leica DMI 5000, while the scanning electron microscope was Tescan Mira3. Aim of this study was to analyse the effect of dispersoid particle concentration on the mechanical performances of the alloy: for this reason image analysis with LAS software has been performed to verify particle distribution. The samples were analysed by means of energy dispersion spectroscopy (EDS) to verify the chemical composition and fracture surfaces were inspected by SEM after bending tests.

## 3. Results and discussion

Different castings showed slightly different compositions. Table 1 shows the average composition of the alloy after casting. This was the mean value obtained by performing EDS analyses on several samples.

After performing preliminary tests with the aim of improving particle distribution in the alloy, reinforced castings have been obtained by using 0.04  $\mu\text{m}$  alumina particles. SEM and optical micrographs of the alloy are shown in Fig. 1. The alloy microstructure is constituted by  $\gamma$  grains and lamellar grains constituted by alternated  $\gamma$  and  $\alpha_2$  phases well visible in Fig. 1a. Fig. 1b clearly shows the presence of alumina agglomerated particles that appear black both in the optical and in the SEM micrographs. Although the average size of the alumina used in the tests was of 0.04  $\mu\text{m}$ , the analyses show that they agglomerate during casting. EDS analyses carried out on these particles allowed to identify

them (Fig. 2). As it can be clearly observed by this figure, agglomerated particles are characterized by different size and shape.

Table 1. Average composition of the TiAl based alloy.

An example of a column heading	Weight (%)	Atom (%)
Al	32.32	46.80
Ti	59.93	48.88
Cr	3.20	2.40
Nb	4.55	1.92

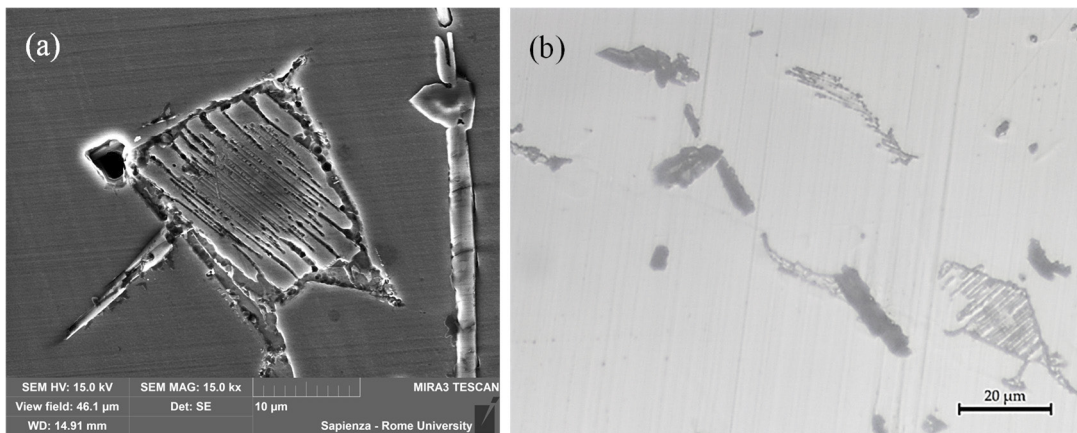


Fig. 1. SEM micrograph(a) and optical micrograph (b) of the produced alloy.

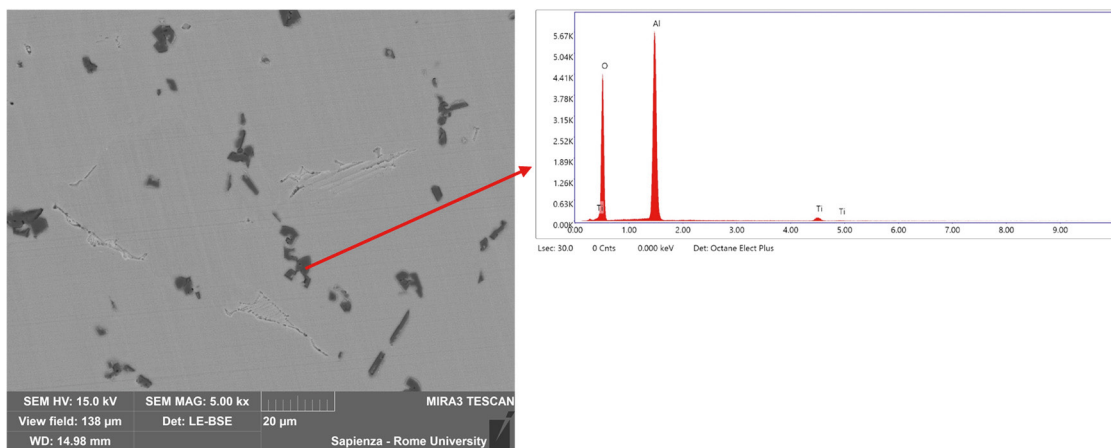


Fig. 2. SEM micrograph of the produced alloy highlighting the EDS analysis of black particles.

Over the last few years different types of dispersoids were tested with good results in terms of mechanical performances. In a previous work the effect of 3%<sub>vol</sub> alumina on TiAl based alloy was studied (Pilone et al. (2020)). In order to compare the results obtained by adding 2%<sub>vol</sub> Al<sub>2</sub>O<sub>3</sub> to the studied alloy image analysis has been performed on several samples. Fig.3 shows the results this analysis.

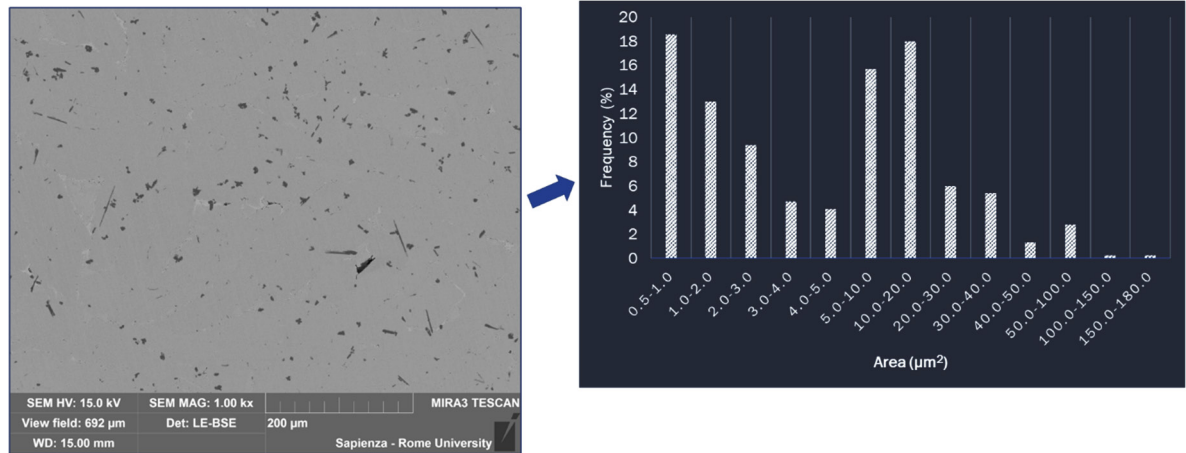


Fig. 3. SEM micrograph of the produced alloy (left) and particle size distribution (right) obtained by image analysis.

Image analysis carried out on some alloy sections revealed that about 60% of particles have a size lower than  $10 \mu\text{m}^2$  and that they are homogeneously distributed throughout the alloy (Fig.3). By comparing these data with the ones found by adding 3%<sub>vol</sub> of alumina it is possible to say that a decrease of alumina percentage in the alloy decreases the degree of particle agglomeration, in fact in the previous research about 65% of alumina particles had a size lower than between 0.5 and  $25 \mu\text{m}^2$  (Pilone et al. (2020)). As highlighted in the previous paper dispersion strengthening is very effective for improving mechanical properties of Ti aluminides. These alloys show a ductile-to-brittle transition temperature that increases by adding dispersoids. At temperatures lower than  $900 \text{ }^\circ\text{C}$  the reinforced alloy has a very brittle behaviour that is very sensitive to the presence of defects like shrinkage defects. If we compare the results at  $900 \text{ }^\circ\text{C}$  we can see (Fig.4) that yield strength increases by adding alumina and that 2%<sub>vol</sub> of  $\text{Al}_2\text{O}_3$  is more effective in increasing strength probably because smaller  $\text{Al}_2\text{O}_3$  particle are able to hinder dislocation motion in a more efficient way. The macrographs in Fig. 4a show that while the base alloy at  $900 \text{ }^\circ\text{C}$  bends without braking (Brotzu et al. (2018)), with 2%<sub>vol</sub>  $\text{Al}_2\text{O}_3$  it fractures after a slight deformation. Then, even the addition of 2%<sub>vol</sub>  $\text{Al}_2\text{O}_3$  determines a more brittle behaviour.

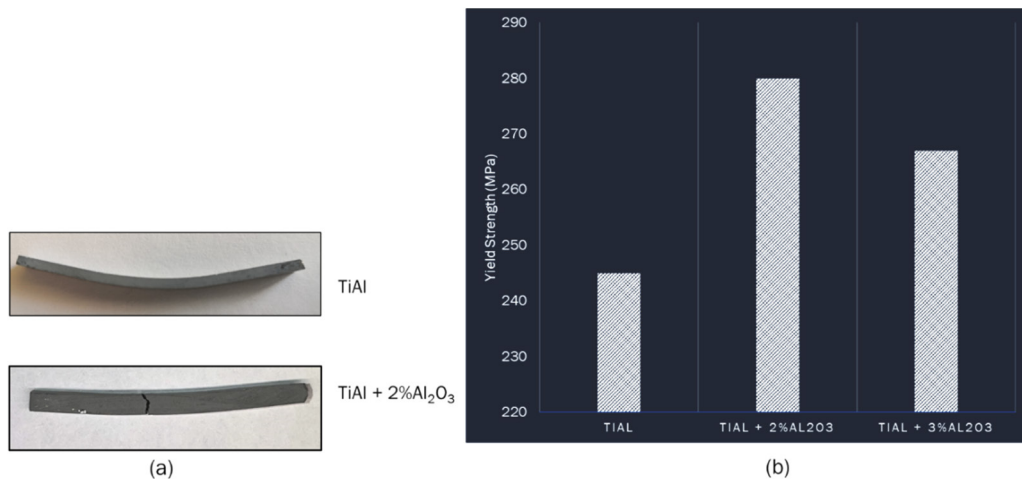


Fig. 4. Macrographs showing the specimens after test at  $900 \text{ }^\circ\text{C}$  (a) and Yield strength of the alloy at  $900 \text{ }^\circ\text{C}$  (b).

If we analyse the effect of added dispersoids on the alloy Young Modulus it is apparent that at 800 and 900 °C it increases when alumina is added to the base alloy and that, again, 2%<sub>vol</sub> Al<sub>2</sub>O<sub>3</sub> is more effective than 3%<sub>vol</sub> due probably to the lower agglomeration degree of dispersoid particles (Fig.5).

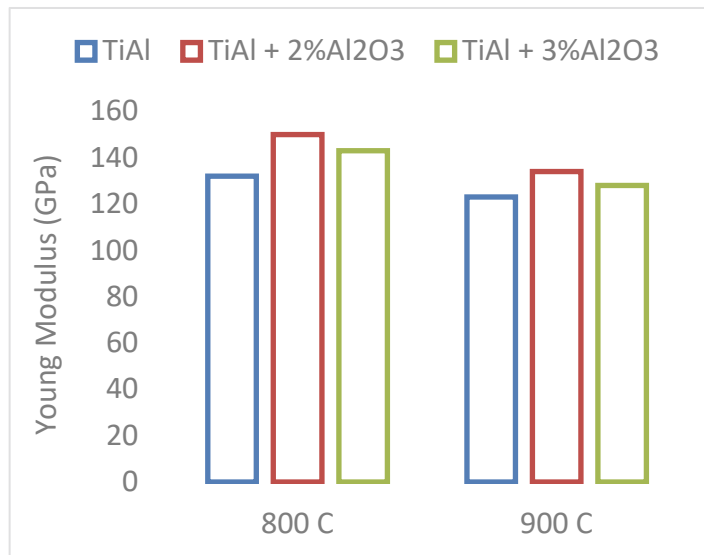


Fig. 5. Young Modulus of the TiAl based alloys at 800 and 900 °C.

Specimens used for bending tests have been produced directly by centrifugal casting and, since they have high surface/volume ratio, several attempts have been done to reduce their tendency to form shrinkage cavities. One of the most critical aspects related to mechanical tests of these alloys is that, being them extremely brittle, tests are very sensitive to defects. Fig. 6 shows two shrinkage cavities found on the fracture surfaces.

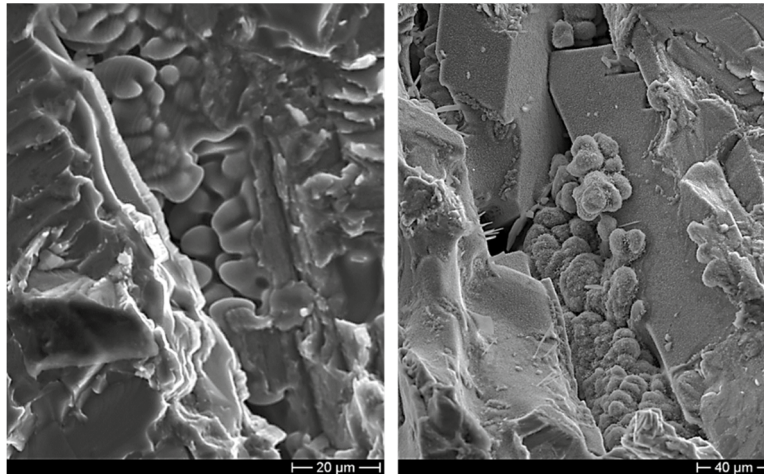


Fig. 6. SEM micrographs showing the presence of shrinkage cavities on fracture surfaces.

SEM analyses carried out on specimen fracture surfaces showed that the fracture propagates following a transgranular path at all the tested temperatures. By observing Figs. 7 and 8 it is possible to see that at 800 and 900 °C the fracture propagates with a mixed mechanism that produces both translamellar and interlamellar fracture. A careful observation of micrographs in Figs.7 and 8 highlights the presence of Al<sub>2</sub>O<sub>3</sub> particles inside lamellar and  $\gamma$  grains. The



observed ceramic particles are not broken and appear well joined to the TiAl matrix. No reinforcements pull-out has been observed

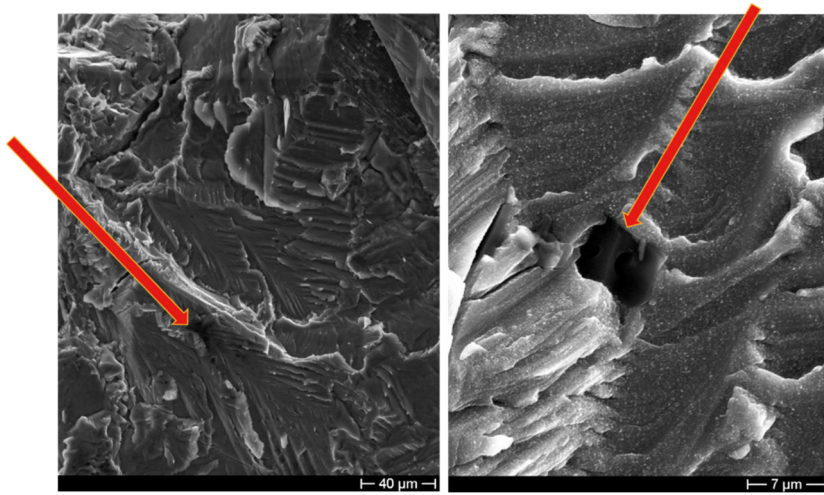


Fig. 7. SEM micrographs showing fracture surface morphology after test at 800 °C. Red arrows indicate  $\text{Al}_2\text{O}_3$  particles.

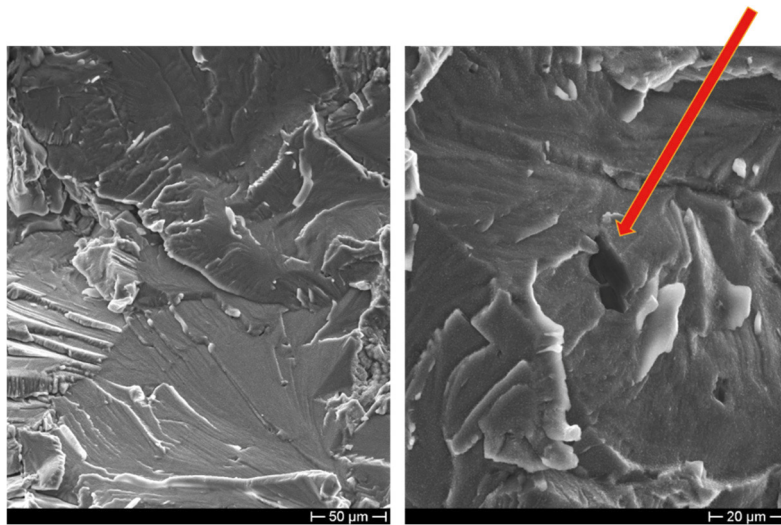


Fig. 8. SEM micrographs showing fracture surface morphology after test at 900 °C. The red arrow indicates an  $\text{Al}_2\text{O}_3$  particle.

#### 4. Conclusions

The main objective of this research was to improve the mechanical properties of TiAl-based alloys by means of dispersion hardening with alumina. The results highlighted that dispersion hardening with 2% alumina seem to be more effective than 3% alumina in increasing the mechanical properties of the alloy at high temperatures, mainly over the 800-900 °C temperature range. The analyses performed on the cast specimens revealed that by decreasing the alumina percentage added to the alloy it is possible to obtain a lower degree of alumina particle agglomeration. This is beneficial in increasing the alloy mechanical properties because smaller particles are more effective in hindering dislocation movement. Further studies are necessary to further improve the dispersion of the nano particles throughout the alloy.

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